



PATENT
85940/15

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Heikkila et al.)	Group Art Unit: 1808
)	
Serial No.: 08/928,893)	Examiner: D. Naff
)	
For: Process for the Simultaneous)	
Production of Xylitol and Ethanol)	
)	
Filed: September 12, 1997)	

Assistant Commissioner for Patents
Washington, D.C. 20231

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DECLARATION UNDER 37 C.F.R. § 1.132
OF HEIKKI HEIKKILA

SIR:

I, Heikki Heikkila, declare as follows:

1. I am currently employed as Process Development Manager for Xyrofin, Oyi and have been since 1994. After receiving my M. Sc. in wood chemistry in 1972 from Helsinki University of Technology, I joined Cultor, S.A. as a research chemist in the area of industrial scale chromatography. In 1981, I became manager of the process development department at Cultor Research Center, and in 1990 I became Development Manager in Finn Sugar Engineering. During the period between 1973 and 1998, my work led to the filing of several patent applications upon which I was named as a co-inventor.

2. I am a co-inventor of U.S. Application Serial No. 08/928,893 filed September 12, 1997. I have reviewed the Examiner's Official Action dated September 16, 1996 in parent Application Serial No. 08/910,133 and the reference cited therein: U.S. Patent No. 5,081,026 ("the Heikkila '026 patent"). I am also a co-inventor of the Heikkila '026 patent.

3. The above-identified application is directed to an efficient process for producing xylitol and ethanol from hexose-rich lignocellulose raw materials with limited waste product. Using the claimed method, the hexose-rich raw material and hexose-rich raw material components may be pre-treated with a hydrolysis step and are fermented such that the hexose present (such as glucose) is converted to ethanol while xylose (a pentose) is converted to xylitol. The ethanol is recovered and the xylitol component is chromatographically separated from the other fermented products, thereby utilizing the main part of the raw material, and also in particular, hexose-rich portions of the raw material, to produce commercially useful amounts of xylitol and ethanol.

4. The Examiner cites the Heikkila '026 patent as disclosing the claimed process, including using starting material containing hexose. The presently claimed process, however, requires the starting material to be "hexose-rich" which is contrary to the teaching of the Heikkila '026 patent. In the Heikkila '026 patent, the raw material has a high xylose content and a low hexose content. For example, in Example 2 of the Heikkila '026 patent, the ratio of xylose to glucose is 21:1. The processing of the raw material, for example, by hydrolysis, is for the purpose of decreasing to the extent of glucose content of the solution. For instance, in Example 2 of the Heikkila '026 patent, the hard wood used, birch wood, has a xylose content which is high. The birch wood is subjected to a mild prehydrolysis to produce a hemicellulose-based prehydrolysate containing a large amount of xylose, a low amount of hexose and small amounts of other components from birch wood and hydrolyses degradation

products. The xylose concentration of the hydrolysate solution is disclosed as being 76 g/l while the glucose (hexose) content is 3.6 g/l. This prehydrolysate rich in xylose is then further processed, e.g., by posthydrolysis, to hydrolyze all oligosaccharides present to monosaccharides, and by fermentation whereby xylose is converted to xylitol and the xylitol produced is recovered. The main part of the product obtained after prehydrolysis, i.e., the solid residue which would contain the hexose component, is not processed nor recovered at all, but rather discarded according to Heikkila '026. Thus, the Heikkila '026 patent does not suggest using a starting material that is rich in hexose or low in xylose content.

5. In contrast to Heikkila '026, according to the present invention xylose and ethanol are produced from raw materials that are high in hexose content. As illustrated in Example 1 of the present specification, the solid residue obtained and discarded in the Heikkila '026 patent is extracted and extensively hydrolyzed as outline at page 9, and xylitol and ethanol are produced and recovered. In the present invention, the bulk of the raw material is processed rather than just the xylose-rich portion as in the Heikkila '026 patent.

6. As an example of the claimed process, fresh Finnish birch chips having the following composition were subjected to acid hydrolysis:

D.S. content (105°C)	66	g/100g
Dissolved solids in acetone	2.6	% of D.S.
Klason-lignin	19.2	% of D.S.
Carbohydrates:		
glucose	41.1	% of D.S.
xylose	21.0	% of D.S.
galactose+rhamnose	1.3	% of D.S.
arabinose	0.4	% of D.S.
mannose	1.5	% of D.S.

The birch chips were dried and 60 g D.S. of the powder was mixed with 600 ml of 72 weight % sulfuric acid. The mixture was digested at 30°C for 1 hour with slow mixing. After the digestion, 16.8 l of distilled water was admixed. The mixture was boiled with reflux for 4

hours. After the boiling, the solution was cooled and filtered in reduced pressure through filter paper. The filtrate obtained had the following composition:

D.S. content	4.4	g/100g
Carbohydrates:		
glucose	3.3	% of D.S.
xylose	1.7	% of D.S.
galactose+rhamnose	0.1	% of D.S.
arabinose+mannose	0.2	% of D.S.
Sulfate	92	% of D.S.

Hexose-rich and xylose-rich fraction is prepared by using chromatographic separation according to U.S. Patent No. 4,631,129. The fermentation of the fraction and the recovery of ethanol and xylitol are carried out as described in the examples in the pending application. Significantly, by this example and using the method of the present application, about 12.5% ethanol and 17.5% xylose on birch wood dry substance are recovered, and only 25% solid waste material is produced.

7. The Examiner also asserts that it would have been obvious to increase the production of ethanol in the method disclosed in the Heikkila '026 patent "by adding glucose to the hydrolyzed starting material or hydrolyzing the starting material with enzymes that produce more glucose." However, there is no suggestion in the Heikkila '026 patent to either utilize the hexose-rich waste product or to separate ethanol as a product of the process.

8. In contrast to the Heikkila '026 patent, the presently claimed process can (1) produce both ethanol and xylitol in high yield, whereas the only product produced by the method of the Heikkila '026 patent is xylitol, (2) produce xylitol when the xylan/xylose content of the raw material is low, whereas the Heikkila '026 patent produces xylitol only when the xylose content of the raw material is high, and (3) better utilize raw material and minimize waste product compared to the Heikkila '026 patent which discards hexose-rich portions.

9. I further declare that all statements made herein of my own knowledge are true, and all statements made of information and belief are believed to be true; further, that the statements were made with the knowledge that willful, false statements and the like so made are punishable by fine, or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Dated: 28.5. 1999

By: Heikki Heikkilä
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